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DIFFUSION OF WATER VAPOUR THROUGH COLD GORE-TEXTM

by

Randall J. Osczevski





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Environmental Protection Section Protective Sciences Division

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ABSTRACT

A simple gravimetric procedure was devised to measure the water vapour diffusion resistance of waterproof, water vapour permeable textiles at temperatures below freezing. The apparent resistance of a hydrophilic film, part of a Gore-TexTM laminate, increased exponentially with a decrease in temperature. At sub-zero temperatures, water vapour transfer through this film was only a few percent of what it is at room temperature. The diffusion process in such materials is discussed. Other hydrophilic films are likely to be similarly affected.

RÉSUMÉ

Nous avons conçu une procédure gravimétrique simple pour mesurer la résistance à la diffusion de vapeur d'eau des textiles perméable à des température sous le point de congélation. La résistance apparente d'un film hydrophile, l'une des composantes d'une doublure Gore-Tex^{MC}, a augmenté exponentiellement avec la baisse de température. Aux températures sous zéro, le transfert de vapeur d'eau à travers ce film n'était qu'à un petit pourcentage de ce qu'il est à la témperature ambiante. Nous présentons dans ce rapport le procédé de diffusion. D'autres films hydrophiles pourraient vraisemblablement être affectés de façon similaire.

EXECUTIVE SUMMARY

A gravimetric method, using ice, was devised to assess the water vapour diffusion resistance of a fabric at a range of temperatures below freezing. The resistance of the hydrophilic component of Gore-TexTM II was measured with the film adjacent to ice. The resistance increased by about an order of magnitude when the temperature was lowered from room temperature to below freezing. Low temperature affects both the free volume in the polymer and the kinetic energy of the molecules of the diffusing vapour.

This experiment clearly demonstrates that large increases in the diffusion resistance of Gore-Tex $^{\rm TM}$ II can be expected at low temperatures. The apparent water vapour diffusion resistance of a hydrophilic, waterproof film or coating depends on its temperature and moisture content. These in turn depend on many factors such as air temperature, wind speed, relative humidity, solar radiation and physical activity. However, generally speaking, there will be little diffusion of moisture through any layer of cold weather clothing that has a waterproof hydrophilic component unless the shell and the vapour diffusing through it are warm.

INTRODUCTION

The water vapour diffusion resistance of a hydrophilic material depends on the relative humidity at which the test is performed (1,2). It has been suggested that the diffusion resistance of some water-vapour permeable coated materials might also depend on temperature (2), as is the case in many other materials (3,4). None of the many methods (5,6,7,8) for determining water vapour diffusion resistance was intended for use below freezing. Therefore a simple gravimetric procedure was devised to test the hypothesis that low temperatures cause a significant increase in the apparent resistance of a hydrophilic material to the diffusion of water vapour.

In very cold or cold windy weather, the inner surface of a windproof shell worn by an active individual commonly becomes coated with ice or frost. The measurements were therefore carried out with one face of the sample in direct contact with ice. This also maximizes the flux of water vapour in the test and defines the relative humidity at one face of the sample.

MATERIALS AND METHOD

Samples

Gore-TexTM II was chosen for investigation because it represents the state of the art in waterproof, water vapour permeable rainwear materials. Gore-TexTM II is a multilayer composite of an outer fabric, a microporous PTFE (TeflonTM) film, a hydrophilic coating and a light inner fabric. Its layers can be peeled apart, leaving the PTFE film attached to the outer fabric and a hydrophilic coating attached to a light nylon tricot (2). The hydrophilic coating is identified by Gore in product advertising as "polyalkylene oxide polyurethane-urea". Three circular samples with a diameter of 8.5 cm were cut from the nylon tricot with this hydrophilic coating. They were conditioned at 65% RH and 20°C. The samples were approximately 0.15 mm thick. The film was probably less than 0.01 mm of this total.

Apparatus

The test cell, shown in Figure 1, had two parts: a glass petri dish and a small commercial plastic container (Polytainer Inc. 403A-8, 225 gram capacity) with a snap-on lid (Polytainer 403 NL). The plastic container, with a circular cutout in its lid, was used to hold the ice and the petri dish to hold the desiccant. The fabric sample was placed between the ice and the desiccant, either directly on the surface of the ice for high humidity tests or on the desiccant bed for low humidity tests.

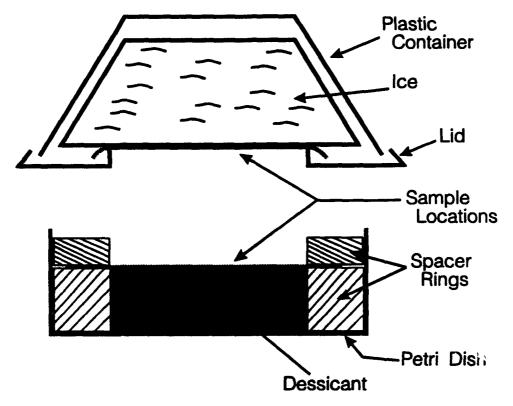


Figure 1. Apparatus for low temperature tests.

Ice Container

Approximately 150 ml of distilled water was frozen in the plastic container, which formed the upper part of the cell. A few millilitres of distilled water were then poured onto the surface of the ice to level it and fill in the inevitable cracks. When this had frozen, the ice was removed from the container and its upper surface was levelled and polished by rubbing it onto a clean, flat surface at room temperature. The ice was then put back into the container which was then placed in a chamber set at the desired test temperature.

In the centre of the lid of the ice container was a circular depression 6.6 cm in diameter and 3 mm deep. The floor of this depression was removed to create a hole for diffusion with an area of 0.0034 m². In use, the container was inverted and the ice sat on the rim of this hole. Theoretically, as ice evaporated over the hole and above the thin rim, the block of ice could settle by almost 3 mm before it contacting the flat area of the lid. This would keep the distance between the ice surface and the desiccant

constant. It was difficult to tell whether this occurred in practice, however, after each experiment there was a sharply defined circular depression in the surface of the disk where ice had evaporated. The depth of the depression was negligible compared to the distance between the ice and the desiccant. The intended separation of the ice and desiccant was 10 mm, however under a load of 150 g, which was equivalent to the mass of the ice, the distance was measured by vernier caliper as 9.5 mm ± 1 mm, at room temperature. At low temperatures, less deflection might be expected under load.

Each sample was placed in the centre of the ice surface with the coating side down and against the ice. A metal disc 5mm thick and slightly smaller in diameter than the hole in the lid was placed on the centre of the sample for a few minutes to flatten it and hold it against the ice. Although the disc was at the test temperature, the sample often bonded to the ice surface while loaded in this fashion. After at least ten minutes the ice container was inverted while the disc, sample and ice were held against the bottom of the container by a finger. The disc, sample and ice were then slowly lowered until the sample and the ice came to rest on the rim, and the disc could be slipped out through the hole. The weight of the ice prevented the sample from slipping past the rim of the hole.

Desiccant Container

The rim of a Pyrex petri dish was sanded flat to remove the glass bead formed in the manufacturing process so that it would conform to the flat surface of the upper part of the cell. A spacer ring of closed-cell foam approximately 9 mm thick, was fitted into the dish. The hole in the ring was the same size as the hole in the lid of the upper part of the cell. This hole was filled with desiccant and the upper surface of the bed was pressed down as flat as possible and level with the top of the foam ring. Eight mesh DrieriteTM, anhydrous CaSO₄, was used as the desiccant. A second ring was used to create a uniform diameter diffusion pathway between the desiccant and the top of the petri dish. A second identical desiccant container was assembled as a conditioning dish.

Procedure

Before each test began, the parts of the cell were kept overnight in the environmental chamber at the appropriate temperature. During this period the upper part of the cell, holding the sample, was placed on the conditioning dish. The lower part of the cell or test dish was covered with a petri dish cover. A half-hour before weighings began, the upper part of the cell was placed on the test dish and the test-dish cover was used on the conditioning dish.

At intervals of two to twelve hours the parts of the cell were separately removed from the chamber and weighed on an electronic scale to the nearest 0.01 g. The two parts of the cell were always weighed in the same order. The dew or frost that formed on the parts of the cells each time they were weighed amounted to less than 10 milligrams when the weighings were quickly performed. As the mass of water that condensed on the cells was approximately the same at each weighing, it cancelled out when the net change was calculated.

The relative humidity in the weighing room was kept below 35% to minimize the frost or dew that formed on the parts of the cell when they were taken out of the chamber. Condensation also formed on everything inside the chamber when its door was opened. This was minimized by reducing the size of the chamber opening. Frost evaporated from the exposed surfaces of the parts of the cell and the petri dish cover between weighings.

While the upper part of the cell was being weighed, the lower part was covered with the petri dish cover from the conditioning dish. When the lower part of the cell was being weighed with this cover in place, the upper part of the cell was in the chamber over the conditioning dish of desiccant. When the cell was reassembled in the environmental chamber, the petri dish cover was placed over the conditioning dish to ensure that the inner surface of the cover was dry when needed for the next weighing.

Changes in the colour of the desiccant, which is blue when dry and pink when saturated, showed that the water vapour was absorbed on the upper surface of the desiccant bed first. No color changes were detectible deeper in the bed until the exposed surface had become completely pink. This occurred when about 0.25 g of water had been absorbed. Experiments were halted before this point was reached. In a few cases, the test was extended by mixing the desiccant to renew the surface. The desiccant in the conditioning dish was mixed or changed at the end of the conditioning period.

Calculations

The total resistance, R, to the diffusion of water vapour between the upper and lower parts of each cell was calculated in the conventional fashion from equation 1:

$$R = A \cdot (P_{ins} - P_{dry}) / M$$
 [1]

where A is the area (m^2) of the sample through which diffusion can occur, P_{ice} is the vapour pressure (pa) over ice at the temperature of the environmental chamber, P_{dy} is the vapour pressure (pa) over the desiccant surface (assumed to be zero) and M (kg/s) is the rate of water vapour transfer between the parts of the cell. The absolute values of the changes in mass of the two sections of the

cell were averaged at each weighing. A linear regression of mass change against time provided the rate of water vapour transfer. Correlation coefficients were always 0.99 or better.

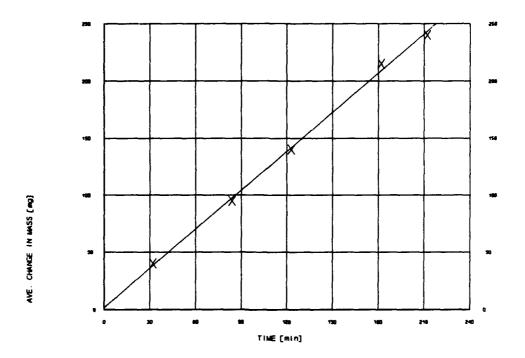


Figure 2. Average change in mass of the two cell parts with sample at -6°C.

Equation [1] gives the total resistance to water vapour diffusion from the ice to the desiccant, which includes the resistance of the air gap. The resistance of air gap was determined by the same procedure, without a sample. From experience with similar materials, the resistance of the knitted backing material on each sample was unlikely to have been more than a few tenths of a millimeter of equivalent standard still air.

RESULTS

The accuracy of the method can be checked by determining the diffusion resistance of the air gap at a range of temperatures. In a dilute mixture of still air and water vapour, the flux of water vapour ϕ is given by Fick's Law (9):

$$\phi = -(D m/R T) (dP/dy)$$
 [2]

where D is the diffusivity of water vapour in air at atmospheric pressure in m^2/s , approximately given (9,10) by:

$$D = 2.5 \times 10^{-5} (T/293)^{1.81}$$
 [3]

and R is the constant in the universal gas law. After integration equation [2] becomes:

$$\phi = -\Delta P/(\ell \cdot R \cdot T/D \cdot m)$$
 [4]

so that the resistivity (m Pa s/kg) is:

$$\rho = \mathbf{R} \cdot \mathbf{T} / \mathbf{D} \cdot \mathbf{m} \tag{5}$$

The length of the diffusion path between the ice and the desiccant can be found by dividing the diffusion resistance by the calculated resistivity of air at the temperature of the experiment. Figure 3 illustrates that for all temperatures, the same path length was found: 10.1 ± 0.5 mm, which agrees nicely with the measured distance between the ice and the desiccant. This agreement confirms that the air in the space was still and that convection was negligible.

In Figure 4, the water vapour diffusion resistances of the samples calculated from the measured fluxes in millimetres of equivalent standard still air (mm ESSA), are plotted against temperature. The resistance increases non-linearly with decreasing temperature from about 3 mm ESSA at -3° C to 26 mm ESSA at -24° C. At -10° C, the water vapour diffusion resistance of the samples on ice averaged about 5 mm ESSA. The diffusion resistance of samples that were placed on the surface of the desiccant at -10° C and not on the ice, was much higher: 69 ± 8 mm ESSA (n=2).

The water vapour permeability of four more samples, cut from the same piece of material was measured at 18° C using an apparatus of the type described by van Beest and Wittgen (8). The samples were placed directly onto the wet surface with the equivalent of a ten-millimetre air gap between the sample and dry air. The diffusion resistance averaged 0.9 \pm 0.5 mm ESSA, which includes a few tenths of a millimetre resistance from the knitted backing material.

In Figure 5, $ln(1/R_{\rm film})$ is plotted against 1/T as suggested by Barrer (3). A straight line with a slope of $-\epsilon/R$ could easily be fitted to this data. The resulting "activation energy", 65 kJ/mole, is surprisingly high compared to other similar materials (4). This is not a real activation energy, for although only the temperature was changed during the experiment, moisture content (regain) of the sample must also have changed. Although at a given relative humidity the equilibrium moisture content of textile materials usually increases with a decrease in temperature, it has

been shown that at sub-zero temperatures the opposite sometimes occurs (11). As the resistance of the material changes more than that of the air gap with which it is in series, the mean relative humidity in the sample must have decreased with temperature.

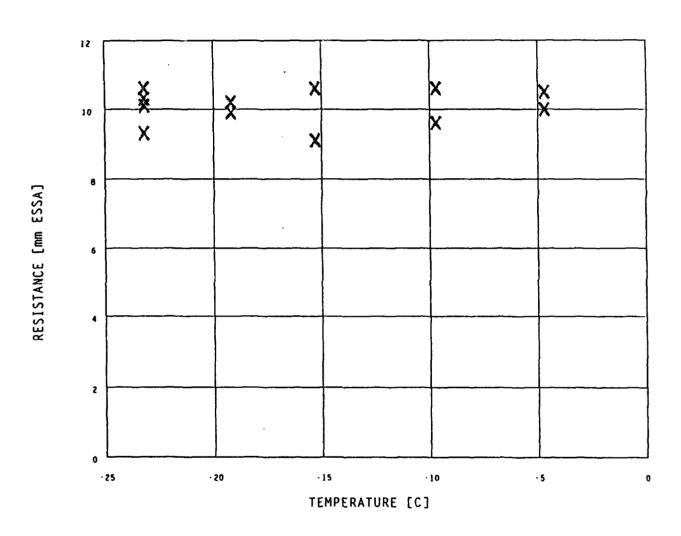


Figure 3. Resistance of the air space in the apparatus.

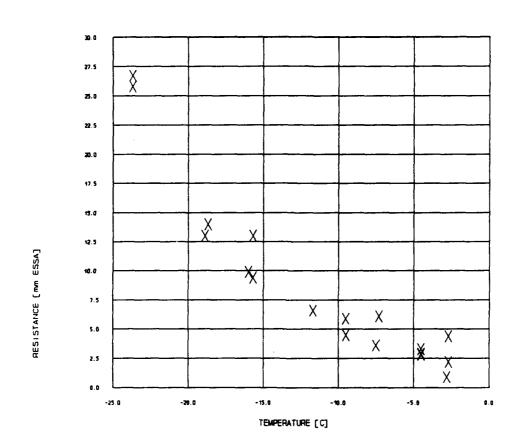


Figure 4. Water vapour diffusion resistance of samples at low temperatures.

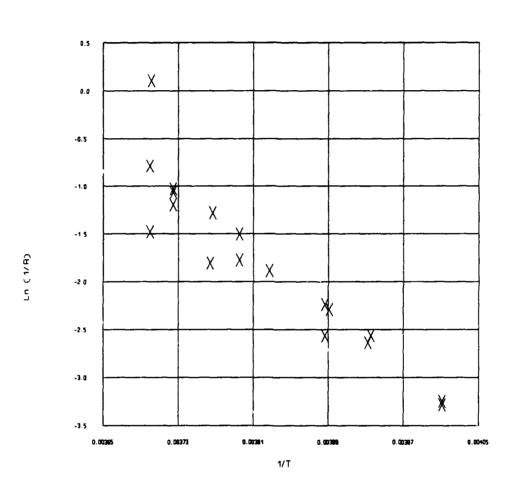


Figure 5. The data replotted in a form suggested by Barrer (3).

DISCUSSION

The sample resistance at any temperature depends to some extent on the thickness of the still air in the apparatus. If the gap had been thinner, a smaller fraction of the total diffusion resistance would have been between the desiccant and the sample. As a result, the relative humidity at the dry face of the sample would have been lower and the diffusion resistance higher because of the lower moisture content of the sample.

The air space simulates the layer of still air that adheres to the outer surface of clothing. The boundary layer is highly susceptible to disturbance by wind and is normally much less than 10 mm thick. An approximate value for the eqivalent thickness of this air layer can be derived from Burton's (12) standard values of the thermal resistance of the ambient air at various wind speeds:

$$\ell = k/((1/R_{th}) - 4\sigma\epsilon \bar{T}^3)$$
 [6]

where k is the thermal conductivity of air in w/mK at \bar{T} , the mean of the air and surface temperatures in degrees Kelvin. R_{th} is the thermal resistance of the air layer (m²K/W), σ is the Stefan-Boltzman constant and ϵ is the effective emissivity, assumed to be 0.9.

In average outdoor calm, the thermal resistance of the air layer can be taken as $0.10~\text{m}^2\text{K/W}$. A wind of 3 m/s reduces the thermal resistance of the boundary air layer (12) to about $0.04~\text{m}^2\text{K/W}$. These resistances are equivalent to air layer thicknesses of 3.7 and 1.1 mm. A ten-millimetre air space was used in the apparatus because the air outside the boundary layer on clothing is never as dry as the air at the surface of the desiccant. In winter, high relative humidity is the norm in many areas (13).

Hydrophilic materials, by definition, absorb water. They have many polar sites which attract and trap molecules of water vapour. The same attraction also allows more energetic molecules of water vapour to diffuse more easily through these materials.

In order to diffuse through a polymeric material a molecule must have enough kinetic energy to push through the spaces between atoms on neighbouring polymer chains. As a molecule pushes into one of these spaces it encounters the electric fields of the atoms comprising the polymer. There is a repulsive interaction with the corresponding fields of the atoms of the diffusing molecule. As the distance between the polymer atoms and the diffusing molecule decreases, the repulsive force increases and an increasing fraction of the kinetic energy of the diffusing molecule is converted to electostatic potential energy. A diffusing molecule with enough initial kinetic energy can squeeze through the gap before all of

its kinetic energy is converted to potential energy. A less energetic molecule would be stopped and then accellerated back in the direction from which it came.

In some ways it is analogous to a golf ball rolling over a rise in a green. As the ball approaches the top of the rise it moves more slowly because some of its kinetic energy is converted to gravitational potential energy. A fast ball can reach the top and, except for friction, regain its speed and kinetic energy while rolling down the other side. A slower ball may fail to surmount the hill and roll back down the way it came. In a polymer, the potential energy landscape across which a water molecule must diffuse is complex. It must forge a route over terrain covered with many hills and dales. Moreover, because of the thermal vibrations of the polymer chains, the distances between atoms on neighbouring chains are always changing and therefore, so are the heights of the potential hills.

Hydrophilic sites in the polymer change the picture considerably. These sites are polar groups, often carbonyl or hydroxyl groups (14) attached to the polymer chain. In simple terms, the electron cloud of the atoms of the group is drawn more strongly towards one of the nuclei of the group. As a result, the nuclear charge of the other atom is only partially shielded and its electrostatic influence can be felt by molecules a large distance away. Because there is an attractive force between the dipole of the water molecule and the polar site their interaction results in a change in potential energy. In the vicinity of a hydrophilic site, a large area of the potential energy "plain" is pulled downwards into a potential well. This lowers the nearby potential hills and allows water molecules with less kinetic energy to diffuse in its vicinity.

If there is another hydrophilic site close by the two potential wells may overlap to create a pass or low energy pathway between sites. When the density of hydrophilic sites is high, continuous low energy pathways may exist from site to site through the material facilitating the diffusion of polar molecules. The height of the passes between potential wells defines the minimum energy a molecule must have to diffuse. This is the activation energy. The greater the density of the attractive sites, the lower the activation energy. If a molecule that has lost energy through a collision has less energy than the activation level, it cannot diffuse any further and is caught in the potential well, i. e., it is absorbed.

The effectiveness of the polar interaction is illustrated by the ease with which water vapour diffuses through Gore-Tex $^{\text{TM}}$ II in contrast to the difficulty that molecules of oxygen and carbon dioxide encounter in the same material. Oxygen and carbon dioxide molecules are about the same size or slightly smaller than water

molecules but they do not have a permanent dipole moment. The resistance of Gore-TexTM II to the diffusion of water vapour at room temperature lies somewhere between 0 and 45 mm ESSA depending on the relative humidity (1,2). In contrast, the resistance of the same material to the diffusion of O_2 at room temperature is orders of magnitude higher, at 8900 mm ESSA (15). Its resistance to CO_2 diffusion is of a similar order (2,15). This relative impermeability to oxygen and carbon dioxide might limit the usefulness of this material in some applications. It is only "breatheable" with respect to water vapour, not respiratory gases.

The moisture content of a hydrophilic polymer also affects its resistance to diffusion. The space between polymer chains in which molecules of water may diffuse, the "free volume", depends to a certain extent on how free the polymer chains are to vibrate. If neighbouring chains are linked together at many points along their length, the molecules will not separate easily and the free volume will be small. Random thermal vibrations will occasionally break one of these links to create a pair of polar sites. If the concentration of water molecules is high, they may become attached to these sites and so prevent the Jink from re-forming. The material is thus plasticized by the water, increasing the free volume. This makes it easier for water molecules to diffuse through the material at high humidity. The attached water molecules can also act as hydrophilic sites to attract othe water molecules.

Because there is less thermal energy at low temperatures the links between chains break less frequently in the cold. Free volume should therefore be smaller at low temperatures. Also, because saturation concentrations of water vapour are much smaller in the cold, we should expect the effect of high relative humidity to be less pronounced. The diffusion resistance and activation energy at any relative humidity might therefore be higher at low temperatures than at room temperature.

The molecules of diffusing vapour are less energetic at lower temperatures. The Maxwell-Boltzman distribution law (16) can be used to calculate the fraction of molecules of a gas with speeds greater than any particular speed c:

$$f_{v>c} = (1 - erf (c/V)) + (2/\pi^{1/2}) \cdot (c/V) \cdot e^{-(c/V)^2}$$
 [7]

where V is the most probable speed, given by:

$$V = (2RT/m)^{\frac{1}{2}}$$
 [8]

m is the mass of a mole of water vapor, 0.018 kg, and erf(x) is the Error Function. For a molecule to have more energy than the activation energy for diffusion, it must have a speed:

The number of molecules having energies greater than the activation energy increases rapidly as the activation energy decreases.

Some of the water molecules in the ice occasionally acquire enough energy to escape the lattice and become vapour. Only a small fraction of these will have enough energy to pass through the narrow spaces between the atoms on adjacent polymer chains. The rest of the vapour will be as irrelevant to the net diffusion rate as the molecules of water that remain trapped in the ice. The flux of water vapour through a hydrophilic material should therefore depend on the difference in the proportion of "activated" molecules on either side of the material, where "activated" is defined as having energies in excess of the activation energy. If $\varepsilon=20$ kJ/mole, according to the Maxwell-Boltzman distribution law the fraction of the total population of vapour molecules that can be considered activated should be 2.4 times greater at 20°C than at -10° C.

In experiments with keratin, King (17) found that the activation energy decreased from 31 kJ/mole at low regain (low moisture content) to 20 kJ/mole at high regain. The lower value is roughly the same as the energy of the hydrogen bond that can form between two molecules of water (18), suggesting that at high moisture content most of the attractive sites may be absorbed molecules of water. The permeability of the keratin in his experiments increased by roughly two orders of magnitude from low to high regain. Equation [7] suggests that if the activation energy for diffusion is 20 kJ/mole rather than 31 kJ/mole, the population of molecules able to diffuse through the polymer should be greater by a factor of seventy-five, which is roughly what was observed.

The concentration of vapour at saturation over water or ice is higher at higher temperatures. Over water at 20°C, it is eight times as high as it is at over ice at -10°C. The maximum flux of water vapour through any clothing layer should therefore be much smaller if it is cold, even if the resistance of the material does not seem to change with temperature. As was shown earlier, at -10°C the fraction of the total population of molecules of water vapour that have enough energy to diffuse through a material is less than one-half of that fraction at room temperature. At -10°C the population is also only about one-eighth what it would be at Considering only these changes in the room temperature. concentration and energy of the vapour, at -10°C we should expect the apparent resistance of the material to be more than twice as large (2.4 times) and the maximum flux of water vapour through the material to be about 5% of the maximum diffusion rate at room This is a somewhat larger flux and lower resistance temperature. than was observed experimentally, indicating that changes in the

material also had some effect.

CONCLUSION

The experiment clearly demonstrates that large increases in the apparent resistance of Gore-TexTMII to water vapour diffusion can be expected at low temperatures. Adjacent to ice at temperatures of -3°C and -24°C, the flux of water vapour through the hydrophilic coating used in Gore-TexTM II was 4% and 0.2% of the flux at room temperature, respectively. It is unlikely that a significant proportion of the water that evaporates from the skin will diffuse through a shell with a hydrophilic component unless the shell and the vapour diffusing through it are warm.

The dipole moment of a water vapour molecule interacts with polar sites on the polymer chain. This allows it to diffuse easily through hydrophilic materials which have many such sites. Non-polar molecules, such as oxygen, have a far harder time. It is likely that the water vapour diffusion resistances of other commercial hydrophilic rainwear materials are also much higher at low temperatures, and that all such materials are also relatively impermeable to non-polar molecules such as oxygen.

Changes in the diffusion resistance of a hydrophilic film are the result of changes in the material and in the vapour diffusing through it. Changes resulting from variations in relative humidity can be qualitatively explained as a result of plasticization of the polymer by water. Changes resulting from variations in temperature probably result from a combination of changes in plasticization and changes in the population of sufficiently energetic vapour molecules. In the cold, there are far fewer molecules of water vapour with enough energy to diffuse through ForeTexTM II.

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A simple procedure was devised to measure the water vapour diffusion resistance of a hydrophilic material at temperatures below freezing. The results indicate that the resistance of the hydrophilic part of a Gore-TexTM laminate is considerably greater in the cold than it is at normal room temperature. The diffusion process in such materials is discussed. It is concluded that the amount of water that can diffuse through any clothing shell, particularly one with a hydrophilic component, may not be significant at temperatures belowfreezing.

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